Potassium in Fertilizers: Volumetric Sodium Tetraphenylboron Method 1

1.0 Scope

Potassium is determined in all types of fertilizers using this method. Some minor modifications are used when analyzing liquid fertilizer samples. Potassium is reported as percent potash (K_2O).

2.0 Summary

The sample is extracted with ammonium oxalate solution. A portion is reacted with excess standard sodium tetraphenylboron solution. The excess sodium tetraphenylboron is titrated with standard benzalkonium chloride solution.

3.0 Comments

When analyzing liquid fertilizers step 6.4 should be omitted from the procedure. The sample may be allowed to stand overnight after step 6.6.

4.0 Apparatus and Materials

4.1 Flask, volumetric, 250 ml, 100 ml, 1L, and 2L

4.2 Glass beads.

4.3 Flask, erlenmeyer, 125 ml, 200 ml and 250 ml.

4.4 Funnels.

4.5 50 ml class A burets.

4.6 10 ml semimicro buret.

4.7 Whatman No. 42 filter paper.

4.8 Hot plate.

5.0 Reagents

5.1 Formaldehyde, 37%.

5.2 Clayton Yellow indicator (0.12%) (Titan yellow, Color Index No. 19540): Dissolve 120 mg of the indicator in 100 ml of deionized water.

5.3 Sodium hydroxide solution (10%): Dilute 100 ml of a 50% solution to 500 ml with deionized water.

5.4 Ammonium oxalate solution (4%): Wear gloves and do not breathe the dust. Add 80 grams of ammonium oxalate to 2 liters of deionized water. Place on a magnetic stirrer to facilitate dissolution.

5.5 Benzalkonium chloride: approximately 0.625% (BAC solution).

5.5.1 Preparation.

A. Dilute 38 ml of 17% Zephiran chloride (Winthrop laboratories; also available at local pharmacies as benzalkonium chloride) to 1 liter with deionized water and mix.

5.5.2 Standardization.

5.5.2.1 Place 1.00 ml of the STPB solution in a 125 ml erlenmeyer flask with about 20-25 ml of deionized water.

5.5.2.2 Add 2 ml of 10% sodium hydroxide, 2.5 ml of formaldehyde, and 1.5 ml of 4% ammonium oxalate solution.

5.5.2.3 Add 6 to 8 drops of the clayton yellow indicator.

5.5.2.4 Titrate with BAC solution to a pink end point using a 10 ml semimicro buret.

5.5.2.5 Adjust the concentration of the BAC so that 2.00 ml = 1.00 ml STPB solution.

5.6 Sodium Tetraphenylboron Solution (STPB): approximately 1.2%.

5.6.1 Preparation.

5.6.1.1 Dissolve 24 g of sodium tetraphenylboron ($NaB(C_6H_5)_4$) in 1600 ml of deionized water with stirring.

5.6.1.2 Add 8 ml of 10% sodium hydroxide and dilute to volume of 2 L with deionized water and mix.

5.6.1.3 Allow the solution to stand for 48 hours.

5.6.2 Standardization.

5.6.2.1 Place about 1.0000 g of potassium dihydrogen phosphate (KH_2PO_4) into a 100 ml volumetric flask.

5.6.2.2 Add 20 ml of 4% ammonium oxalate solution and dilute to volume with deionized water and mix.

5.6.2.3 Transfer three 15 ml aliquots of the solution to three 100 ml volumetric flasks.

5.6.2.4 Add 4 ml of 10% sodium hydroxide, 5 ml of formaldehyde, and 43.00 ml of STPB to each flask.

5.6.2.5 Dilute to volume with deionized water and mix.

5.6.2.6 Allow the solutions to stand for 5-10 minutes and then pass through dry filter (Whatman #42).

5.6.2.7 Pipet a 50 ml aliquot of the filtrate into 125 ml erlenmeyer flasks and add 6 to 8 drops of clayton yellow indicator solution.

5.6.2.8 Titrate the solutions with adjusted BAC solution.

5.6.2.9 Calculate F with the following equation for each titration:

$$F = \frac{(34.61)(\text{wt. } KH_2PO_4)}{(43 \text{ ml STPB} - \text{ml BAC})} = \%\ K_20/\text{ml STPB}$$

5.6.2.10 Calculate an average F and record.

5.6.2.11 How 34.61 was arrived at for a number.

$$\frac{(1.0000 \text{ g } KH_2PO_4)(1 \text{ mole } KH_2PO_4)(1 \text{ mole } K_20)(94.186 \text{ g } K_20)}{} = 0.3461 \text{ g } K_20$$

$(136.051\ g\ KH_2PO_4)(2\ mol\ KH_2PO_4)(1\ mol\ K_20)$

$\frac{0.3461\ g\ K_20}{1.0000\ g\ KH_2PO_4}$ = (0.3461)(100)= 34.61 when diluted to 100 ml.

6.0 Procedure

6.1 Weigh about 2.5 g of sample (1.25 g if the potash claim is greater than 50%) and wash through a funnel with deionized water into a 250 ml volumetric flask. Record the weight of the sample to the nearest 0.0001 g.

6.2 Add approximately 125 ml of deionized water and 4 glass beads to the flask.

6.3 Add 50 ml of the 4% ammonium oxalate solution.

6.4 Boil the sample for 30 minutes on a hot plate and then cool the solution. If organic matter is present, add 2 g of K-free charcoal before boiling.

6.5 Dilute the solution to volume with deionized water and mix well.

6.6 Let the solution stand until clear or pass through a dry filter before proceeding.

6.7 Transfer a 15 ml aliquot of the sample solution to a 100 ml volumetric flask.

6.8 Add 4 ml of 10% sodium hydroxide and 5 ml of 37% formaldehyde.

6.9 Fill a clean 50 ml buret with the standard STPB solution.

6.10 Add standard STPB solution to the 100 ml volumetric flask from the buret. The ml added should be equal to the potash claim plus five ml. Record the ml added. (Add ml = 1/2 claim plus 5 ml if 1.25 g sample was used.)

6.11 Dilute to volume with deionized water, mix thoroughly and allow to stand for 5-10 minutes.

6.12 Filter the solution through 15 cm Whatman No. 42 filter paper into a 250 ml erlenmeyer flask. Do not wet the filter paper or rinse the flasks.

6.13 Transfer 50 ml of the filtrate to a 125 ml erlenmeyer flask and add 6-8 drops of the Clayton Yellow indicator.

6.14 Using the standard BAC, titrate the sample solution to a pink end point. Record

the ml of BAC used.

7.0 Calculations

7.1 The percent potash (K_20) is calculated according to the following equation:

$$\% \text{ Potash} = \frac{(\text{ml STPB} - \text{ml BAC}) \times F \times 2.5}{\text{(sample weight)}}$$

Where: ml STPB = ml added in step 6.10
ml BAC = ml used in step 6.14

F = Factor from STPB standardization

Sample weight = weight recorded in step 6.1

8.0 Quality Control

8.1 Standardization of the STPB and BAC solutions.

8.1.1 Weigh the primary standard to exactly 1.0000 g and record.

8.1.2 Record all titration data.

8.1.3 Adjust BAC concentration so that 2.00 ml = 1.00 ml STPB.

8.1.4 Record all calculated titers (F) and calculate an average.

8.2 Monitor time.

8.2.1 Let prepared STPB reagent stand at least 48 hours before standardizing.

8.2.2 Boil samples in Ammonium oxalate solution for 30 minutes.

8.2.3 After STPB is added to precipitate the K and the solution is brought to volume and mixed thoroughly, it should stand at least five minutes before filtering.

9.0 Bibliography

Official Methods of Analysis (1984) 14th Ed., AOAC, Washington, D.C., secs. 2.119-2.121